

N-Heterocyclic Carbene-Catalyzed Oxidation of Unactivated Aldehydes to Esters

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General Information

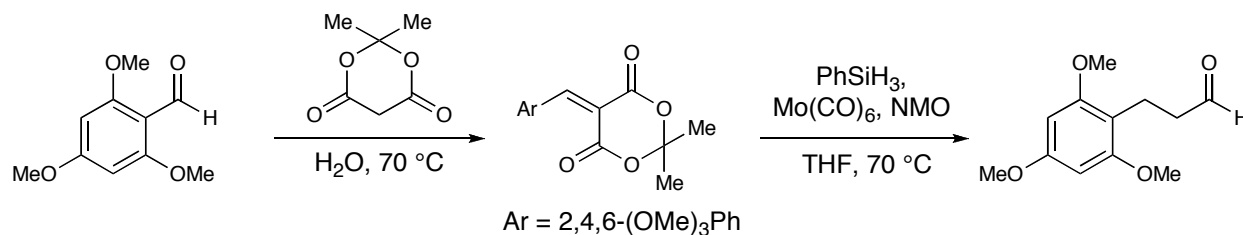
All reactions were carried out under a nitrogen atmosphere in flame-dried glassware with magnetic stirring. Toluene, MeOH, and CH₂Cl₂ were purified by passage through a bed of activated alumina.¹ Reagents were purified prior to use unless otherwise stated following the guidelines of Perrin and Armarego.² Purification of reaction products was carried out by flash chromatography using EM Reagent silica gel 60 (230-400 mesh). Analytical thin layer chromatography was performed on EM Reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light and anisaldehyde, ceric ammonium nitrate stain, potassium permanganate, or phosphomolybdic acid followed by heating. Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. ¹H-NMR spectra were recorded on a Varian Inova 500 (500 MHz) or Mercury 400 (400 MHz) spectrometer and are reported in ppm using

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1. Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometal.* **1996**, *15*, 1518-1520.
 2. Perrin, D. D. and Armarego, W. L. *Purification of Laboratory Chemicals*; 3rd Ed., Pergamon Press, Oxford. 1988.

solvent as an internal standard (CDCl_3 at 7.26 ppm). Data are reported as (ap = apparent, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad; coupling constant(s) in Hz; integration. Proton-decoupled ^{13}C -NMR spectra were recorded on a Varian Inova 500 (125 MHz) or Mercury 400 (100 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl_3 at 77.0 ppm). Mass spectra data were obtained on a Varian 1200 Quadrupole Mass Spectrometer and Micromass Quadro II Spectrometer.

Aldehydes were purchased from Sigma-Aldrich unless otherwise noted and distilled before use. Alcohols were likewise purchased from Sigma-Aldrich. 1,4-dimethyltriazolium iodide was synthesized from 1-methyltriazole following the procedure of Miyashita.³ Tetrabutylammonium persulfate was prepared from commercial Oxone (Sigma-Aldrich) using the method of Trost.⁴ Manganese(IV) oxide and all other oxidants were commercially available from Sigma-Aldrich Chemical Company.

Synthesis of Aldehyde Substrates



3-(2,4,6-Trimethoxyphenyl)propanal was synthesized following a slight modification of the method of Frost⁵ 2,4,6-trimethoxybenzaldehyde (3.0 g, 15.3 mmol, purchased from Sigma-Aldrich) and 2,2-dimethyl-1,3-dioxane-4,6-dione (Meldrum's acid, 2.42 g, 16.8 mmol, 1.1 equiv, purchased from Sigma-Aldrich) were combined in H_2O (30 mL) and heated at 70 °C for 3 hours. The precipitate was collected by filtration and triturated with warm methanol to give a yellow solid (5-(2,4,6-trimethoxybenzylidene)-2,2-dimethyl-1,3-dioxane-4,6-dione, 4.17 g, 85% yield). The methanol washings could be concentrated and the resulting solid recrystallized from methanol to obtain further condensation product.

In a flask outfitted with a water-cooled condenser, 5-(2,4,6-trimethoxybenzylidene)-2,2-dimethyl-1,3-dioxane-4,6-dione (1.61 g, 5.0 mmol) was stirred in THF (30 mL) with *N*-methylmorpholine *N*-oxide (59 mg, 0.50 mmol, 10 mol %) and molybdenumhexacarbonyl (66 mg, 0.25 mmol, 5 mol %). To this mixture was added phenylsilane (1.85 mL, 15.0 mmol, 3 equiv) and the reaction was heated at 70 °C overnight (monitored by TLC, 7:3 hexanes/ethyl

3. Miyashita, A.; Suzuki, Y.; Nagasaki, I.; Ishiguro, C.; Iwamoto, K.; Higashino, T. *Chem. Pharm. Bull.* **1997**, 45, 1254-1258.
4. Trost, B. M.; Braslau, R. *J. Org. Chem.* **1988**, 53, 532-537.
5. Frost, C. G.; Hartley, B. C. *Org. Lett.* **2007**, 9, 4259-4261.

acetate). After cooling to room temp. 6 mL H₂O was added and stirred for 10-15 minutes. The mixture was diluted with diethyl ether and washed with 1M NaOH (3x) and brine (2x). The aqueous layers were combined and extracted with Et₂O. The organic layers were combined, dried with MgSO₄, and concentrated in vacuo. The residue was purified by column chromatography (15% ethyl acetate/hexanes) to give the desired aldehyde (346 mg, 31% yield) as a yellow oil.

R_f = 0.57 (7:3 hexanes/ethyl acetate);

¹H NMR (500 MHz, CDCl₃) δ 9.75 (s, 1H); 6.12 (s, 2H); 3.80 (s, 3H); 3.78 (s, 6H); 2.91 (t, *J*=7.6 Hz, 2H); 2.53 (t, *J*=7.6 Hz, 2H);

¹³C NMR (125 MHz, CDCl₃) δ 204.04, 159.85, 158.75, 108.90, 90.49, 55.65, 55.44, 43.70, 15.99;

IR (film); 3055, 2941, 2839, 2720, 1720, 1265.

LRMS (APCI): Mass calculated for C₁₂H₁₆O₄ [M+H]⁺ 225.1. Found 225.0.

Other aldehydes were prepared in an analogous manner. Spectral properties for **3-(1-methyl-indol-3-yl)propanal**,⁶ **3-(2-furyl)propanal**,⁷ and **3-(2-thiophenyl)propanal**⁸ matched those found in the literature.

3-tert-butyldimethylsiloxy-2-methylpropanal,⁹ **3-(2-pyridyl)propanal**,¹⁰ **3-(tert-butyldimethylsiloxy)propanal**,¹¹ and **3-(tert-butyldiphenylsiloxy)propanal**¹² were prepared by oxidation of the corresponding alcohol. Spectral properties matched those found in the literature.

General Procedure for triazolium-catalyzed oxidation

A flame-dried round bottom flask was charged with triazolium salt (11 mg, 0.05 mmol). The flask was sealed with a septum and put under positive pressure of nitrogen. Dichloromethane (2.5 mL) and DBU (82 μL, 0.55 mmol) were added followed by the aldehyde (0.5 mmol). The septum was removed and to the flask was added MnO₂ (217 mg, 2.5 mmol). The flask was then sealed with septum and methanol (0.100 mL, 2.5 mmol) was added *via* syringe. The reaction stirred at ambient temperature until aldehyde was consumed (monitored by GC or TLC). The mixture was filtered through a thin pad of silica, which was washed with ethyl acetate (15 ml). The filtrate was then concentrated *in vacuo*. The resulting residue was purified by flash chromatography on silica gel.

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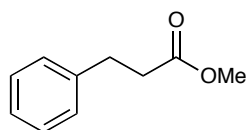
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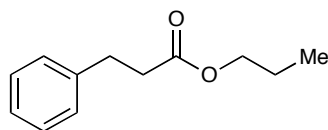
10. Kitbunnadaj, R.; Zuiderveld, O. P.; Christophe, B.; Hulscher, S.; Menge, W. M. P. B.; Gelens, E.; Snip, E.; Bakker, R. A.; Celanire, S.; Gillard, M.; Talaga, P.; Timmerman, H.; Leurs, R. *J. Med. Chem.* **2004**, 47, 2414-2417.

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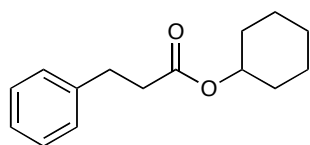
12. Heumann, L. V.; Keck, G. E. *Org. Lett.* **2007**, 9, 1951-1954.



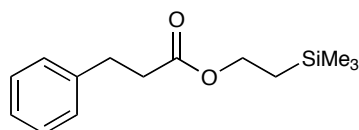
Methyl 3-phenylpropionate (6): Filtration yielded 81 mg (98%) of methyl 3-phenylpropionate as a yellow oil. Spectral data matched those found in the literature.¹³ When 19 mmol (2.5 mL) of hydrocinnamaldehyde was employed with 10 mol % triazolium salt (420 mg, 1.9 mmol), 1.1 equiv. DBU (3.13 mL, 20.9 mmol), and 5 equiv. of MnO₂ (8.26 g, 95 mmol) and methanol (4.1 mL, 95 mmol) in 95 mL of CH₂Cl₂, the ester was obtained in 98% yield (3.08 g).



n-Propyl 3-phenylpropionate (7): Purification by column chromatography (10% ethyl acetate/hexanes) yielded 79 mg (82%) of *n*-propyl 3-phenylpropionate as a yellow oil. Spectral data matched those found in the literature.¹⁴



c-Hexyl 3-phenylpropionate (8): Purification by column chromatography (8% ethyl acetate/hexanes) yielded 102 mg (88%) of *c*-hexyl 3-phenylpropionate as a yellow oil. Spectral data matched those found in the literature.¹⁵



2-trimethylsilylethyl 3-phenylpropionate (9): Purification by column chromatography (8% ethyl acetate/hexanes) yielded 106 mg (85%) of 2-trimethylsilylethyl 3-phenylpropionate as a yellow oil.

R_f = 0.79 (4:1 hexanes/ethyl acetate)

¹H NMR (500 MHz, CDCl₃) δ 7.28-7.31 (m, 2H); 7.20-7.22 (m, 3H); 4.17 (t, *J* = 8.5 Hz, 2H); 2.95 (t, *J* = 7.8 Hz, 2H); 2.61 (t, *J* = 7.8 Hz, 2H); 0.97 (t, *J* = 8.5 Hz, 2H); 0.04 (s, 9H);

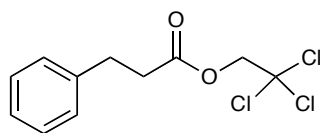
¹³C NMR (125 MHz, CDCl₃) δ 173.3, 140.8, 128.7, 128.5, 126.4, 62.9, 36.3, 31.2, 17.5, -1.3;

LRMS (APCI): Mass calculated for C₁₄H₂₂O₂Si [M+H]⁺ 250.1. Found 250.0

13. Mantecon, S.; Vaquero, J. J.; Alvarez-Builla, J.; de la Puente, M. L.; Espinosa, J. F.; Ezquerra, J. *Org. Lett.* **2003**, 5, 3791-3794.

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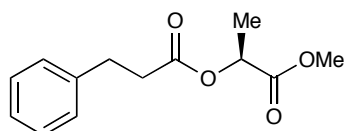
2,2,2-trichloroethyl 3-phenylpropionate (10): Purification by column chromatography (8% ethyl acetate/hexanes) yielded 122 mg (87%) of 2,2,2-trichloroethyl 3-phenylpropionate as a yellow oil.

R_f = 0.63 (4:1 hexanes/ethyl acetate)

^1H NMR (500 MHz, CDCl_3) δ 7.20-7.34 (m, 5H); 4.74 (s, 2H); 3.03 (t, J = 7.8 Hz, 2H); 2.81 (t, J = 7.8 Hz, 2H)

^{13}C NMR (125 MHz, CDCl_3) δ 171.5, 140.1, 128.8, 128.5, 126.7, 93.9, 74.2, 35.7, 30.9;

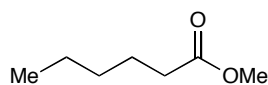
LRMS (APCI): Mass calculated for $\text{C}_{11}\text{H}_{11}\text{Cl}_3\text{O}_2$ $[\text{M}+\text{H}]^+$ 280.0. Found 280.1



Methyl (*S*)-2-(3-phenylpropyloxy)propionate (11): Purification by column chromatography (5% ethyl acetate/hexanes) yielded 87 mg (74%) of Methyl (*S*)-2-(3-phenylpropyloxy)propionate as a light yellow oil. Spectral data matched those found in literature.¹⁶ Enantiomeric ratio was determined by HPLC (Chiralcel OD-H, 1.5% *i*-PrOH/Hexanes) Retention times – 11.05, 12.30.

^1H NMR (500 MHz, CDCl_3) δ 7.31-7.20 (m, 5H); 5.10 (q, J = 7.0, 1H); 3.74 (s, 3H); 2.98 (t, J = 7.9, 2H); 2.75-2.70 (m, 2H); 1.47 (d, J = 7.3, 3H);

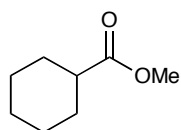
^{13}C NMR (125 MHz, CDCl_3) δ 172.5, 171.5, 140.5, 128.7, 128.5, 126.5, 68.7, 52.5, 35.7, 30.9, 17.1;



Methyl hexanoate (12): Filtration yielded 59 mg (91%) of methyl hexanoate as a light yellow oil. Spectral data matched those found in literature.¹⁷

16. Chan, A.; Scheidt, K. A. *Org. Lett.* **2005**, 7, 905-908.

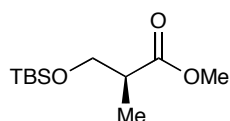
17. Rhee, H.; Kim, J. Y. *Tetrahedron Lett.* **1998**, 39, 1365-1368.



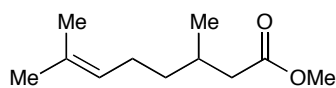
Methyl cyclohexylcarboxylate (13): Filtration yielded 68 mg (96%) of methyl cyclohexylcarboxylate as a yellow oil. Spectral data matched those found in the literature.¹⁸

¹H NMR (500 MHz, CDCl₃) δ 3.65 (s, 3H); 2.29 (m, 1H); 1.88 (dd, 2H); 1.74 (dd, 2H); 1.63 (m, 1H); 1.42 (m, 2H); 1.25 (m, 3H);

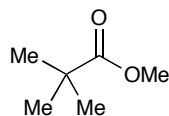
¹³C NMR (125 MHz, CDCl₃) δ 176.75, 51.59, 43.25, 29.16, 25.88, 25.68;



Methyl (S)-3-tert-butyldimethylsiloxy-2-methylpropionate (14): Standard procedure with 70 mg (0.35 mmol) of aldehyde and 20 mg 1,4-dimethyl triazolium iodide (0.088 mmol, 25 mol %), (0.39 mmol, 1.1 equiv.), 150 mg MnO₂ (1.75 mmol, 5 equiv.), and 70 μL methanol (1.75 mmol, 5 equiv.) in 1.75 mL dichloromethane. Purification by column chromatography (2% ethyl acetate/hexanes) yielded 74 mg (91%) of methyl (S)-3-tert-butyldimethylsiloxy-2-methylpropionate as a colorless oil in 92% ee. Spectral data matched those found in the literature.¹⁹ Enantiomeric ratio was determined by HPLC (Chiralcel OD-H, 1.5% *i*-PrOH/Hexanes) Retention times: 4.00, 4.86.



Methyl citronellate (15): Filtration yielded 86 mg (93%) of methyl citronellate as a yellow oil. Spectral data matched those found in the literature.²⁰



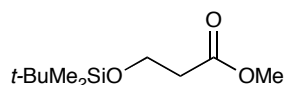
Methyl pivalate (16): Purification by column chromatography (2% ethyl acetate/hexanes) yielded 33 mg (56%) of methyl pivalate as a colorless oil. Spectral data matched those found in the literature.²¹

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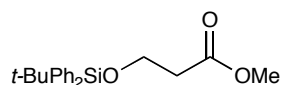
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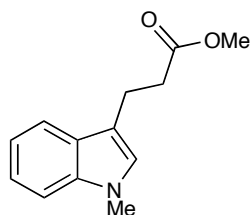
Methyl 3-(*tert*-butyldimethylsiloxy)propionate (17): Filtration yielded 102 mg (94%) of methyl 3-(*tert*-butyldimethylsiloxy)propionate as a yellow oil. Spectral data matched those found in the literature.²²

¹H NMR (500 MHz, CDCl₃) δ 3.89 (t, 2H); 3.67 (s, 3H); 2.53 (t, 2H); 0.87 (s, 9H); 0.05 (s, 6H);
¹³C NMR (125 MHz, CDCl₃) δ 172.37, 59.18, 51.62, 38.01, 25.92, 18.34, -5.28;



Methyl 3-(*tert*-butyldiphenylsiloxy)propionate (18): From 78 mg (0.25 mmol) of aldehyde, filtration yielded 81 mg (95%) of methyl 3-(*tert*-butyldiphenylsiloxy)propionate as a yellow oil. Spectral data matched those found in the literature.²³

¹H NMR (500 MHz, CDCl₃) δ 7.75 (m, 2H); 7.70 (m, 2H); 7.41 (m, 6H); 3.98 (t, *J*=6.3 Hz, 2H); 3.71 (s, 3H); 2.60 (t, *J*=6.3 Hz, 2H); 1.11 (s, 9H);
¹³C NMR (125 MHz, CDCl₃) δ 172.38, 135.66, 134.92, 129.73, 127.80, 59.95, 51.69, 37.81, 26.69, 19.13;



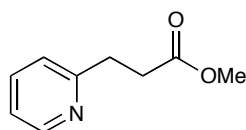
Methyl 3-(1-methyl-indol-3-yl)propionate (19): From 50 mg (0.27 mmol) of aldehyde, filtration yielded 52 mg (90%) of methyl 3-(1-methyl-indol-3-yl)propionate as a yellow oil. Spectral data matched those found in the literature.²⁴

¹H NMR (500 MHz, CDCl₃) δ 7.62 (d, 1H); 7.31 (d, 1H); 7.27 (d, 1H); 7.16 (m, 1H); 6.89 (s, 1H); 3.75 (s, 3H); 3.71 (s, 3H); 3.13 (t, *J*=7.8 Hz, 2H); 2.75 (t, *J*=7.8 Hz, 2H);
¹³C NMR (125 MHz, CDCl₃) δ 173.94, 137.06, 127.62, 126.37, 121.67, 118.85, 118.83, 113.46, 109.30, 51.65, 35.06, 32.66, 20.62;

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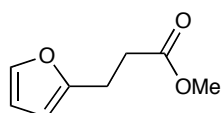
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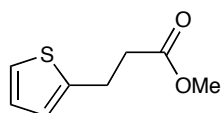
Methyl 3-(2-pyridyl)propionate (20): Filtration yielded 73 mg (88%) of methyl 3-(2-pyridyl)propionate as a yellow oil. Spectral data matched those found in the literature.²⁵

¹H NMR (500 MHz, CDCl₃) δ 8.54 (d, 1H); 7.61 (t, 1H); 7.20 (d, 1H); 7.13 (m, 1H); 3.69 (s, 3H); 3.13 (t, *J*=7.8 Hz, 2H); 2.83 (t, *J*=7.8 Hz, 2H);

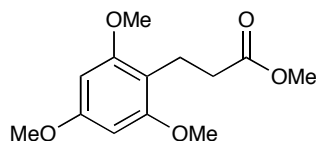
¹³C NMR (125 MHz, CDCl₃) δ 173.73, 160.16, 149.50, 136.57, 123.18, 121.55, 51.82, 33.34, 33.04



Methyl 3-(2-furyl)propionate (21): From 29 mg (0.23 mmol) of aldehyde, filtration yielded 33 mg (92%) of methyl 3-(2-furyl)propionate as a yellow oil. Spectral data matched those found in the literature.²⁶



Methyl 3-(2-thiophenyl)propionate (22): Filtration yielded 82 mg (96%) of methyl 3-(2-thiophenyl)propionate as a yellow oil. Spectral data matched those found in the literature.²⁷



Methyl 3-(2,4,6-trimethoxyphenyl)propionate (23): From 90 mg (0.4 mmol) of aldehyde, filtration yielded 101 mg (99%) of methyl 3-(2,4,6-trimethoxyphenyl)propionate as a colorless oil. Spectral data matched those found in the literature.²⁸

¹H NMR (500 MHz, CDCl₃) δ 6.11 (s, 2H); 3.80 (s, 3H); 3.78 (s, 6H); 3.67 (s, 3H); 2.90 (t, *J*=8.2 Hz, 2H); 2.45 (t, *J*=8.2 Hz, 2H);

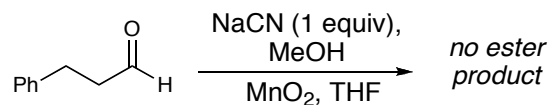
¹³C NMR (125 MHz, CDCl₃) δ 174.38, 159.71, 158.88, 109.29, 90.45, 55.67, 55.38, 51.48, 33.81, 18.50;

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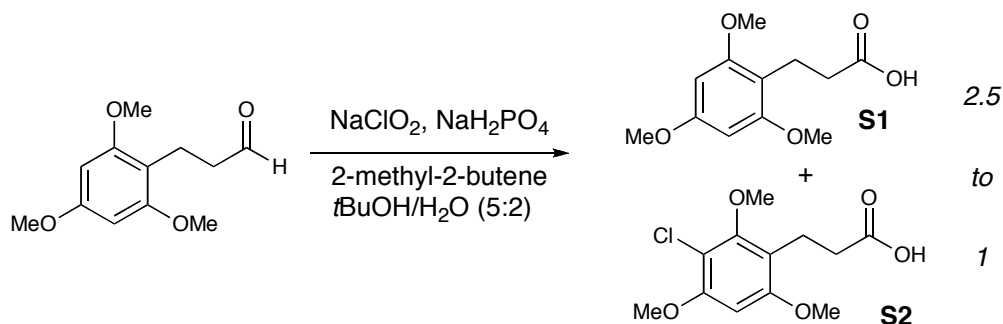
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28. Shi, Z. J.; He, C. J. *Org. Chem.* **2004**, 69, 3669-3671.

Attempted Cyanide-Mediated Oxidation of Hydrocinnamaldehyde

Following the procedure of Taylor,²⁹ a flame-dried round bottom flask was sealed with a septum and put under positive pressure of nitrogen. Tetrahydrofuran (5 mL) was added followed by hydrocinnamaldehyde (66 μL , 0.5 mmol) and sodium cyanide (24 mg, 0.5 mmol). The septum was removed and to the flask was added MnO_2 (650 mg, 7.5 mmol). The flask was then sealed with septum and methanol (0.100 mL, 2.5 mmol) was added *via* syringe. The reaction was stirred at ambient temperature and monitored by GC or TLC. No methyl ester product was observed (by GC, TLC, or ^1H NMR) and the aldehyde starting material remained after 12 hours of reaction time.

Pinnick Oxidation of 3-(2,4,6-trimethoxyphenyl)propanal

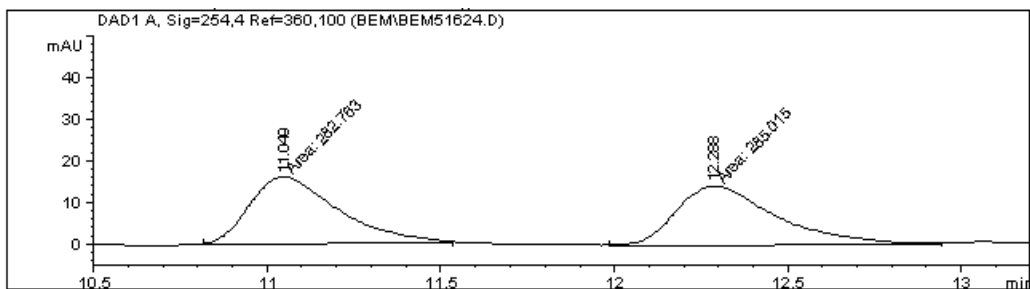
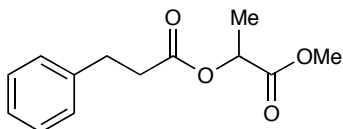
A flame-dried round bottom flask was charged with aldehyde (56 mg, 0.25 mmol) and sealed with a septum and put under positive pressure of nitrogen. A 5:2 mixture of *t*-butanol and water (1.0 mL) was added and the mixture was cooled to 0 °C. 2-Methyl-2-butene (66 μL , 0.625 mmol), $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ (86 mg, 0.625 mmol), and sodium chlorite (80%) (70 mg, 0.625 mmol) were added sequentially and stirring was continued at 0 °C for 1.5 hours. The reaction was warmed to ambient temperature and allowed to stir for an additional 1.5 hours, then quenched with ammonium chloride, diluted with dichloromethane and extracted with dichloromethane (3 x 10mL) and ethyl acetate (1 x 10mL). The organic layers were combined, dried with Na_2SO_4 , and concentrated. ^1H NMR of the residue showed a mixture of products corresponding to 3-(2,4,6-trimethoxyphenyl)propionic acid (**S1**) and 3-(3-chloro-2,4,6-trimethoxyphenyl)propionic acid (**S2**) in a 2.5:1 mixture favoring **S1** based on integration of the protons attached to the aromatic rings; **S1**: δ 6.12 (2H); **S2**: δ 6.05 (1H).

LRMS (APCI): Mass calculated for **S1**, $\text{C}_{12}\text{H}_{16}\text{O}_5$ $[\text{M}+\text{H}]^+$ 241.1. Found 241.0. Mass calculated for **S2**, $\text{C}_{12}\text{H}_{15}\text{O}_5\text{Cl}$ $[\text{M}+\text{H}]^+$ 275.1. Found 274.8.

29. Foot, J. S.; Kanno, H.; Giblin, G. M. P.; Taylor, R. J. K. *Synthesis* **2003**, 1055-1064.

HPLC Traces

Methyl (±)-2-(3-phenylpropanoxy)propionate

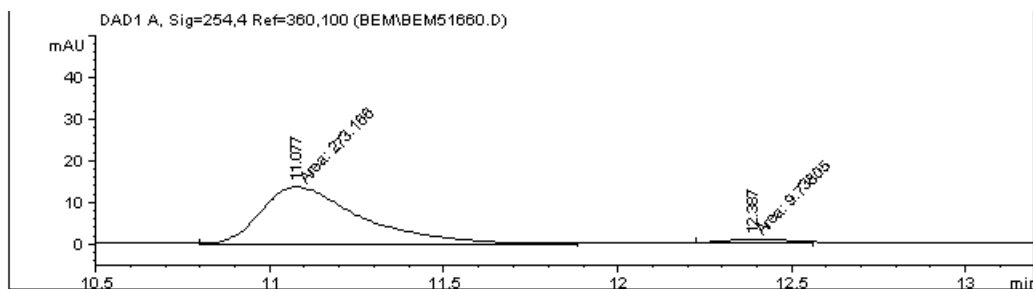
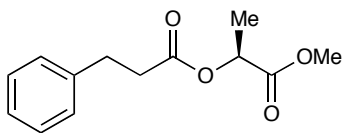


Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

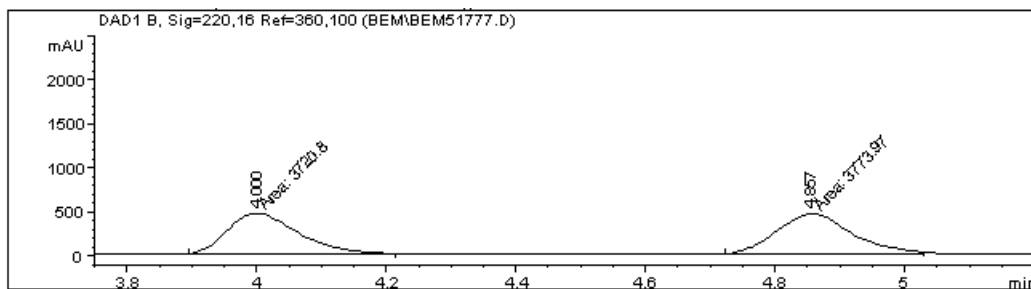
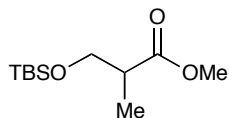
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.049	MM	0.2953	282.76303	15.95690	49.8017
2	12.288	MM	0.3361	285.01453	14.13506	50.1983

Methyl (S)-2-(3-phenylpropyloxy)propionate=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

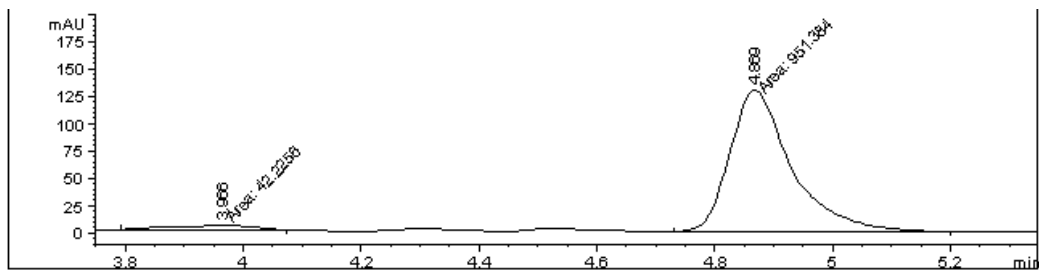
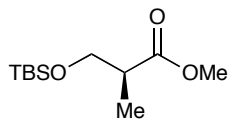
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.077	MM	0.3334	273.16592	13.65366	96.5578
2	12.387	MM	0.2137	9.73805	7.59320e-1	3.4422

Methyl (±)-3-*tert*-butyldimethylsiloxy-2-methylpropionate=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.000	MM	0.1319	3720.80127	470.28741	49.6453
2	4.857	MM	0.1367	3773.96680	460.11865	50.3547

Methyl (S)-3-*tert*-butyldimethylsiloxy-2-methylpropionate=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.966	MM	0.1528	42.22564	4.60604	4.2497
2	4.869	MM	0.1216	951.38373	130.42247	95.7503

NMR Spectra of New Compounds

